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14. ABSTRACT

Burn rate modifiers for solid propellants are being developed based on reactive materials prepared by Arrested Reactive Milling. The materials include nanocomposite thermite compositions using aluminum as a fuel and oxides of iron, molybdenum and copper as oxidizers. The materials are characterized using a set of laboratory tests. Conditions for preparation of viable propellant additives are selected based on the reduced ignition temperature and accelerated burn rate achieved for the prepared nanocomposite powders. The compositions addressed in this study are metal rich, minimizing the energetic penalty due to adding these materials to the propellant formulations. Propellants are formulated with selected samples and their mechanical and burn properties are assessed. Processing reactive nanocomposite powders with conventional propellant formulations was successful and mechanical properties were acceptable. Strand combustion experiments and subscale ballistic evaluations showed substantial increase in the burn rate over a broad range of pressures achieved with replacement of only 5 % of aluminum in aluminized propellant with new materials. Furthermore, the slope break in the log of burn rate vs. log of pressure curve is shifted to a higher pressure for the formulation with the nanocomposite modifier. This shift is expected to be advantageous for the design of rocket motors operating at pressures higher than those used for the current high performance aluminized HTPB propellants.

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SOLID PROPELLANT BURN RATE MODIFIERS BASED ON REACTIVE NANOCOMPOSITE MATERIALS PREPARED BY ARRESTED REACTIVE MILLING

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Abstract

Burn rate modifiers for solid propellants are being developed based on reactive materials prepared by Arrested Reactive Milling. The materials include nanocomposite thermite compositions using aluminum as a fuel and oxides of iron, molybdenum and copper as oxidizers. The materials are characterized using a set of laboratory tests. Conditions for preparation of viable propellant additives are selected based on the reduced ignition temperature and accelerated burn rate achieved for the prepared nanocomposite powders. The compositions addressed in this study are metal rich, minimizing the energetic penalty due to adding these materials to the propellant formulations. Propellants are formulated with selected samples and their mechanical and burn properties are assessed. Processing reactive nanocomposite powders with conventional propellant formulations was successful and mechanical properties were acceptable. Strand combustion experiments and subscale ballistic evaluations showed substantial increase in the burn rate over a broad range of pressures achieved with replacement of only 5 % of aluminum in aluminized propellant with new materials. Furthermore, the slope break in the log of burn rate vs. log of pressure curve is shifted to a higher pressure for the formulation with the nanocomposite modifier. This shift is expected to be advantageous for the design of rocket motors operating at pressures higher than those used for the current high performance aluminized HTPB propellants.

Introduction

Metallized propellants are extensively used for both large and small scale propulsion systems. While aluminum added to propellant formulations brings a number of benefits including reduced combustion instability, increased specific impulse, and increased burn rate [1], the completeness of Al combustion may be low. This is primarily due to aluminum particle agglomeration prior to their ignition [2, 3]. Significant efforts were made recently to understand and control the agglomeration processes [4, 5]. Alleviation of this problem holds significant promise to improve efficiency of aluminum combustion, enable better control over the propellant burn rate, and potentially achieve higher specific impulse.

This effort explores a new approach to improve performance of aluminized propellants by adding fully dense nanocomposite reactive materials as burn rate modifiers. Such modifiers ignite at temperatures substantially lower than characteristic temperature of aluminum ignition. The combustion temperatures of the modifiers are high, so that once such powders are ignited, they assist in rapid heating and ignition of neighboring aluminum particles. In addition to conductive and convective heat transfer from ignited burn rate modifier particles to aluminum, several other heat transfer mechanisms are being enabled to accelerate ignition. Because of high particle combustion temperatures, radiation becomes a significant heat transfer mechanism. Release and condensation of transient gas species accompanying combustion of nanocomposite particles accelerates heat transfer as well. It also improves the convective heat transfer rate and minimizes agglomeration of unignited Al particles.

The increased reactivity and reduced ignition temperatures of the new burn rate modifiers are due to their chemical composition and microstructure. These materials include two or more components capable of highly exothermic reaction. Such components are mixed on the nanoscale so that the exothermic reaction can occur very rapidly. Unlike conventional nanopowder mixtures, these materials consist of fully dense, micron-sized particles with bulk properties similar to those of pure Al powder. Therefore, many concerns associated with nanopowders, including their high cost, difficulty of processing and mixing, reduced energetic content, and environmental and health issues do not apply.

The manufacturing process, Arrested Reactive Milling [6], or ARM, is readily scalable and involves high energy ball milling. The fully dense, micron-sized nanocomposite powders are produced using a top-down manufacturing approach, starting from commercially available micron-scale powders of individual components, e.g., elemental Al and Fe_2O_3 or other metal oxide powders. Particle sizes of the starting powders are not critical, and using very fine or nanosized powders as starting materials is, in fact, undesirable. The nanocomposite material is produced by mechanical milling, which is stopped (or arrested) when the material structure is reduced to the nanoscale but before the chemical reaction is mechanically triggered [6 – 16]. During the milling, formation of nano-scale structures by repeated deformation and shear of starting powder particles is continuously accompanied by agglomeration and cold welding of newly formed particles, so the final powder has particles with dimensions of the order of 1 – 100 μm. Each such particle is a fully dense nanocomposite. It has been observed that essentially any combination of reactive materials can be processed by ARM to prepare a nanocomposite energetic powder. Table 1 lists all the compositions prepared and tested to date including multiple thermites, gas-generators, and metal-metalloid types of reactive components. Each composition is characterized by its unique adiabatic flame temperature. Some of the compositions produce limited quantities of gas. Some of the gases, such as copper vapor, are transient and exist only at high temperatures, while others, such as nitrogen, can contribute to an increase in static pressure.

Table 1. Reactive nanomaterials prepared to date

Nanocomposite Thermites (stoichiometric and other compositions)							
Fuel	Oxidizer						
	Fe_2O_3	MoO_3	CuO	Bi_2O_3	WO_3	SrO_2	$NaNO_3$
Al	x	x*	x*	x	x	x	x**
Mg		x	x				x
$Al_{0.5}Mg_{0.5}$							x
MgH_2		x	x				
Si		x	x	x			
Zr		x	x	x			x
2B·Ti***							x**

Reactive Metal-Metalloid composites	
B	Reactive metals: Ti, Zr, Hf

Nanostructured Al-based alloys	
Al	Alloying components: W, Hf, Mg, MgH_2, Ti, Li, Zr, C

* Metal-rich nanocomposites also have been synthesized

** Metal-lean nanocomposites also have been synthesized

*** Nanocomposite powder

Recently, effectiveness of B-Ti nanocomposite additives prepared by ARM in reducing Al agglomeration and accelerating the propellant burn rate was observed in experiments using a window bomb [17]. This effort develops different nanocomposite powder additives to be used as burn rate modifiers for HTPB propellants.

Materials

Three material compositions were prepared for initial tests; all materials used Al as a fuel with MoO_3, CuO, and Fe_2O_3 used as oxidizers. Starting materials used for preparing nanocomposites were: aluminum powder, -325 Mesh, 99.5% purity, by Atlantic Equipment Engineers; MoO_3 (99.95% purity) and Fe_2O_3 (99.5% purity) powders by Alfa Aesar, and CuO powder (99 % purity, 25µm) by Sigma Aldrich. For each composition, aluminum concentration was two times greater than required for the stoichiometric thermite.

The nanocomposite materials were prepared by ARM [6] using a Retsch PM 400 planetary mill operated at 350 rpm. Hardened steel milling balls, 3/8” diameter were used. Ball to powder mass ratio was 3. Hexane was used as a process control agent. An air conditioner was installed to accelerate the cooling of milling vials.

A set of preliminary experiments was performed to identify appropriate milling times for each material. The materials prepared under different conditions were examined using a scanning electron microscope and ignited in a small closed container to determine the generated pressure pulse. X-ray diffraction (XRD) patterns were also taken, which showed that the prepared composites selected for further tests contained no detectable

reaction products. An example of the XRD pattern is shown in Fig. 1 for $4Al \cdot Fe_2O_3$ nanocomposite.

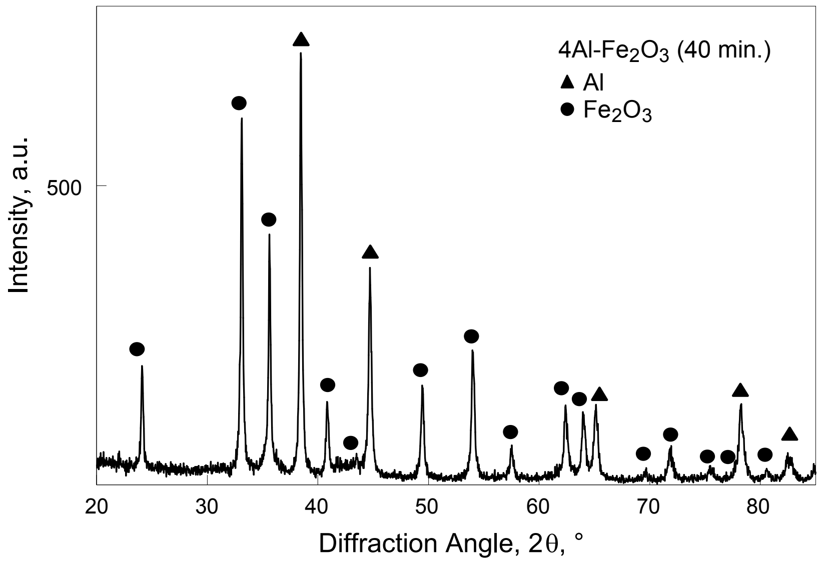

Fig. 1. X-ray diffraction pattern for the prepared $4Al \cdot Fe_2O_3$ nanocomposite powder. Only peaks of starting materials, Al and Fe_2O_3 are detected.

The powders contained equiaxial particles with dimensions varied around 2 – 10 µm. A scanning electron microscope (SEM) image of the prepared $4Al \cdot Fe_2O_3$ nanocomposite powder is shown in Fig. 2.   Figure 2a shows an overview of the particle shapes and morphologies. The images are taken using a backscattered electron detector, so that the brightness is affected by the atomic weight of the materials. The particles appear uniform in brightness indicating a very uniform mixing between Al and Fe_2O_3. A few bright spots can be noticed, these can be assigned to iron particles formed as a result of the thermite reaction occurring locally during the milling. Such thermite reaction is undesirable but is difficult to avoid completely. Figure 2b shows a close-up to the largest formation found in this sample that was clearly produced as a result of such a reaction. The large spherical particle is Al_2O_3, and bright metallic iron particles are attached to it.

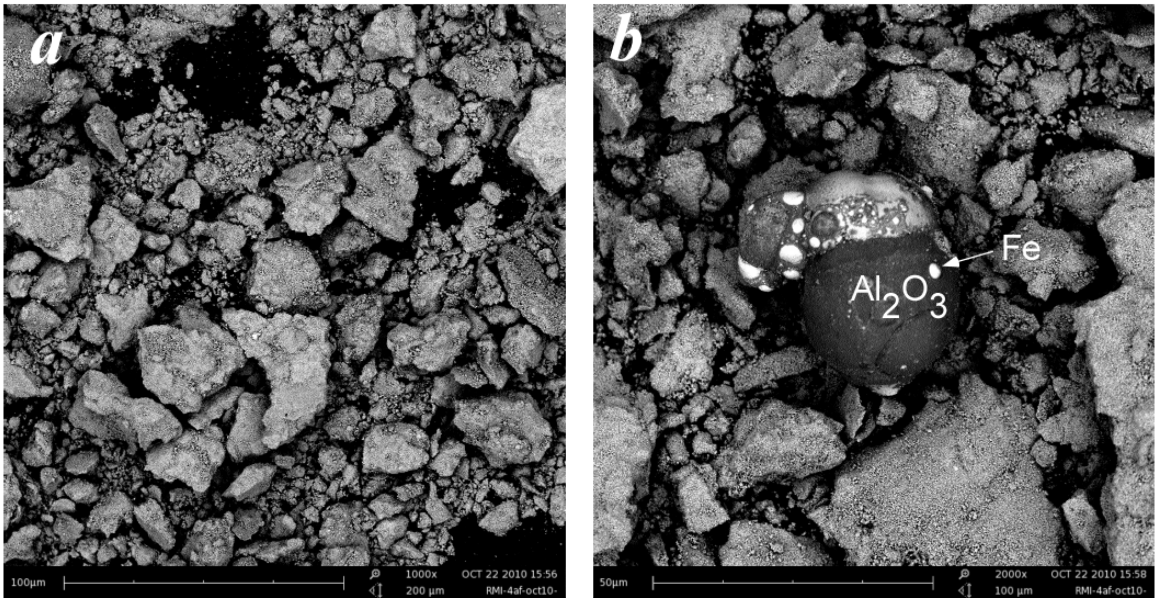

Fig. 2. An SEM image of the prepared $4Al{\cdot}Fe_2O_3$ nanocomposite powder. *a*. Overview. *b*. Close-up of a reacted particle found in the sample

Exothermic reactions in the prepared powders were studied using differential scanning calorimetry (DSC). A characteristic DSC trace for the $4Al{\cdot}Fe_2O_3$ nanocomposite powder is shown in Fig. 3.

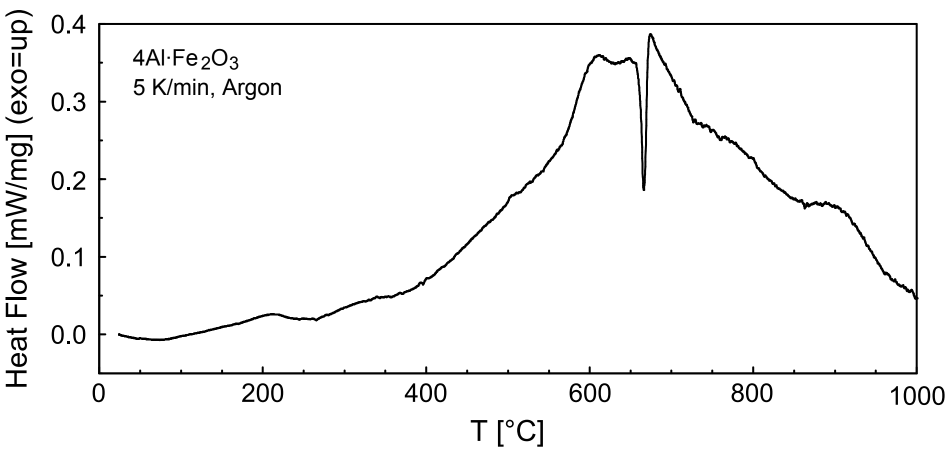

Fig. 3. DSC trace measured for the $4Al{\cdot}Fe_2O_3$ nanocomposite powder heated at 5 K/min.

A broad exothermic event begins at very low temperatures, but becomes strong at about 400 ºC. The reaction is likely composed of many overlapped individual steps, controlling by release of oxygen as Fe_2O_3 decomposes into Fe_3O_4, FeO, and eventually forming metallic Fe.

To simplify implementing the potential burn rate modifiers into HTPB propellant formulations, immediately after preparation, all powders were mixed to form a 50/50 wt % slurry with the R45M hydroxyl terminated polybutadiene pre-polymer used to form the propellant binder. Compatibility of the prepared nanocomposite powders with R45M

was characterized using differential scanning calorimetry (DSC). The slurries were heated up to 300 ºC with no significant exothermic reactions detected.

Initial characterization of propellant mixes

Prepared slurries were characterized for compatibility with different propellant ingredients using differential thermal analysis. In particular, compatibility of all prepared slurries with each individual HTPB propellant ingredient, including ammonium perchlorate, was established. Materials were considered compatible if they did not depress the first exotherm of the control material by greater than 15% per degree. Finally, slurries were used to prepare propellant formulations. An HTPB propellant containing 20% aluminum was also prepared for baseline comparisons. This formulation will herein be referred to as baseline aluminized HTPB. In propellants with nanocomposite additives, a fraction of Al was replaced with the nanocomposite powder.

Mixes containing $4Al \cdot Fe_2O_3$ and $4Al \cdot CuO$ nanocomposites were manufactured and cured with no issues. However, a mix containing $4Al \cdot MoO_3$ nanocomposite agglomerated to form an almost dry, granular mass. Therefore, the latter mix was not considered for further testing. In addition to the above mixes and the reference propellant, a formulation containing an off-the shelf iron oxide as a burn rate modifier was also prepared. Respective propellant mix is referred to as 1BPC-7324. Viscosities for each prepared mix (excluding 1BPC-7324) are shown in Table 2. EOM refers to "End of Mix" and +1 and +2 indicate time in hours from end of mix.

Table 2. Haake 20 Rheometer viscosities of the propellant mixes prepared for the strand burner tests

Mix # (1BPC-)	Thermite Type	% Thermite	Viscosity (High Shear)			Viscosity (Low Shear)		
			EOM	EOM+1	EOM+2	EOM	EOM+1	EOM+2
7316	None	0.0	2.79	2.77	2.87	8.24	12.28	11.58
7317	$4Al \cdot Fe_2O_3$	5.0	5.42	6.59	7.78	62.11	39.44	38.54
7318	$4Al \cdot CuO$	5.0	4.98	5.60	6.83	15.58	19.53	22.44
7323	$4Al \cdot Fe_2O_3$	6.446	4.55	5.37	6.31	15.57	20.18	18.73

Viscosities of the propellant mixes are increased with addition of potential burn rate modifiers, however, that increase is within the range that can be readily handled by formulators. As an example, an increase in concentration of the $4Al \cdot Fe_2O_3$ nanocomposite from 5 to 6.45% actually resulted in a small reduction of the mix viscosity, which is most likely related to particle packing, was observed.

Prepared mixes were used to manufacture propellant strands that were then burned in a Crawford-type bomb [18] to determine burning rates. The burn rates were measured and are shown in Fig. 4. A significant increase in the burn rate is observed for the thermite containing formulations compared to the baseline formulation, 1BPC-7316. The difference in the burn rates is only marginal between 1BPC-7317 and 1BPC-7323 containing 5% and 6.446% of nanocomposite $4Al \cdot Fe_2O_3$. Interestingly, the burn rate is also substantially increased by simply adding iron oxide. Thus, the burn rate

measurements should be complemented with thrust measurements for which the improvement would signify a more efficient aluminum combustion.

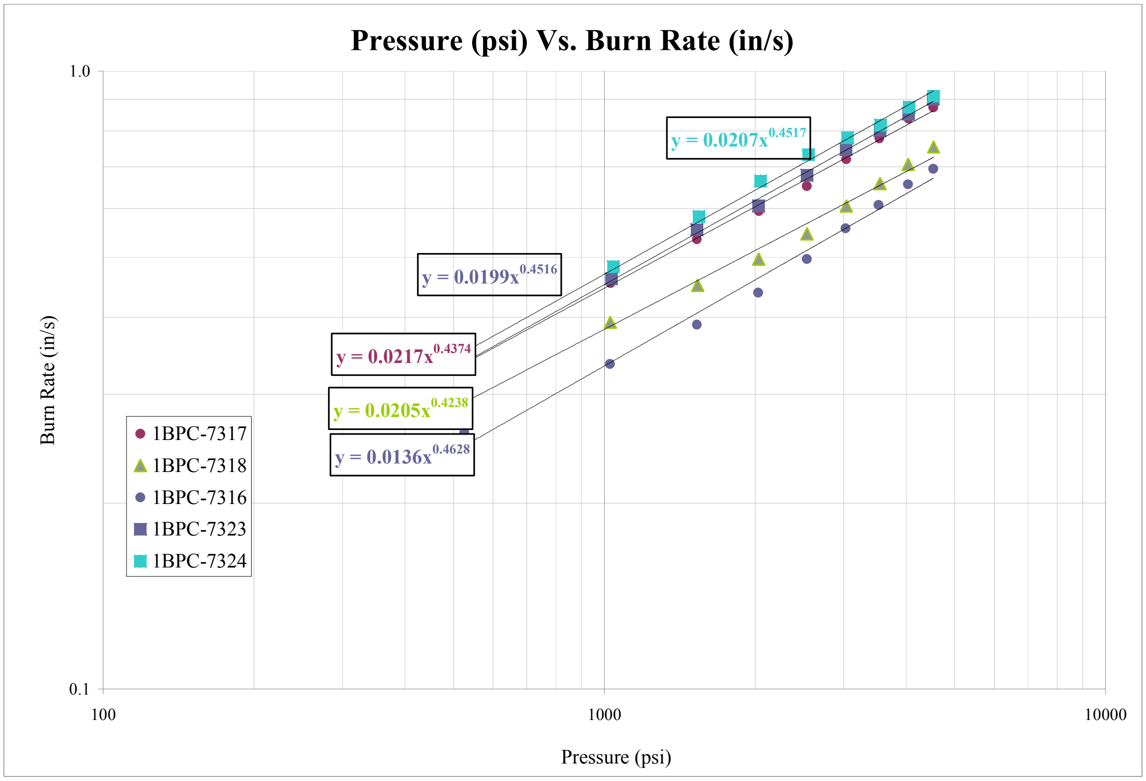

Fig. 4. Pressure-Rate Data for Control and Experimental Propellants

Subscale ballistic evaluation

Based on the assessment of a combination of properties, including mechanical properties of the prepared propellant, strand burning tests, and observed curing details for the propellants with nanocomposite additives, it was decided to select the formulation with 5% of the $4Al{\cdot}Fe_2O_3$ thermite for further tests and evaluations.

A new batch of $4Al{\cdot}Fe_2O_3$ was manufactured and new propellant mixes containing the above additive were prepared for subscale ballistic tests. The viscosities of the new mixtures are presented in Table 3 and are within the limits of processability at 5% weight loading.

Table 3. Viscosity of the propellant mixes prepared for the subscale ballistic evaluations

Mix # (10BPC-)	Thermite Type	% Thermite	Viscosity (High Shear)			Viscosity (Low Shear)		
			EOM	EOM+1	EOM+2	EOM	EOM+1	EOM+2
5035	None	0.0	2.21	2.33	2.47	3.41	4.23	3.77
5036	$4Al \cdot Fe_2O_3$	5.0	6.13	7.03	8.02	24.47	23.89	29.18

Safety evaluation of the prepared propellant formulation (designated as SAA-157) was performed and is documented in Table 4.

The purpose of the friction tests is to evaluate the friction sensitivity of energetic substances. Fixed steel wheel slides over sample on steel plate. The wheel is lowered onto the sample and adjusted to obtain desired pressure. A pendulum is raised to the height for the desired speed. When the pendulum falls, it strikes the plate holder causing it to move 1-inch under the fixed wheel. Test starts at 8 fps. and 800 lbs. Trials are performed until 20 "No-fires" are recorded in a row. The progression of weight from 800 lbs is: 660, 560, 420, 370, 320, 240, 180, 130, 100, 50 and, 25 lbs. If 20 "No go's" are not recorded at 8 fps. and 25 lbs, the test is run at 6 fps. and 800 lbs. From there, the weight is again decreased. If necessary, rates can be decreased to 4, 3, and 2 fps. A "Go" is defined as: Noise greater than that produced by collision of the machine components, flash or sparking, smoke, and flame traces on the machine components

The purpose of the impact test is to determine drop weight impact sensitivity of energetic, explosive materials. The ABL Impact Test uses a 2 kg weight released by an electromagnet, with a drop height from 1 to 80 cm. Weight impacts hardened steel striker, which in turn impacts the sample resting on a hardened steel anvil. The contact surfaces are hardened steel, made of specific steel with a specified roughness. "Success" or "Go" recording indicated by smoke, sparks, or ignition. "Fail" or "No go" recording for no ignition indications. The test finds the highest level with zero goes in 20 trials. When "Go" is reported within 20 drops, height is decreased until level is reached where 20 tests are conducted with "No go" results

Table 4. Evaluation of the prepared propellants

Propellant designation	Comment/Reason for Test	Impact, cm	Friction, ft/s (8 lbf)	ESD, mJ
AHOPS (uncured)	Uncured minimum requirement	11	24	75
SAA-157 (uncured)	Scale-up	17	96	1320
AHOPS (cured)	Cured minimum requirement	21	24	75
SAA-157 (cured)	Scale-up	21	160	266

It was established that SAA-157 meets all AHOPS (ATK's Hazard Policy for the limits for handling/manufacturing energetic formulations) minimum values, and it was approved for scale-up.

In order to size throat diameters for the 2x4 test series, the 2x4's were modeled using Allegany Ballistic Lab's in-house ballistics code, Zeus89, which is a computational tool for zero-dimensional performance analysis. Strain free grain geometry was used with the

grain analysis code P64109 to develop a surface burn back profile. Thermo-chemical inputs were determined from the NASA-Lewis code CET86, which assumes them to be functions of chamber pressure. Using these parameters, the throat diameters were sized to give the desired average pressures to characterize the burning rate curve of the propellant.

The prepared propellant and sub-scale ballistic motors were placed into cure for 7 days at 333 K (60 ºC). Once cured, the motors were disassembled from the cast/cure tooling for build-up in the static fire test arrangement. The motors were conditioned for 4 h at 294 K (21 ºC) before successful static fire of all ten motors.

Pre-and post-fire predictions and measurements are shown in Tables 5 and 6 for the baseline aluminized HTPB propellant and SAA-157, respectively.

Table 5. Pre-and post-fire predictions and measurements baseline aluminized HTPB propellant at 294 K (21 ºC)

Throat diameter, inches	Prediction		Measured		
	P_{ave}, psi	r, inch/s	P_{max}, psi	P_{ave}, psi	R, inch/s
0.373	503	0.270	653	614	0.298
0.321	1006	0.344	985	931	0.339
0.271	1508	0.396	1631	1448	0.391
0.246	2024	0.445	2264	1874	0.433
0.216	3003	0.549	3734	2454	0.490

Table 6. Pre-and post-fire predictions and measurements SAA-157 propellant at 294 K (21 ºC)

Throat diameter, inches	Prediction		Measured		
	P_{ave}, psi	r, inch/s	P_{max}, psi	P_{ave}, psi	R, inch/s
0.433	503	0.361	604	567	0.375
0.373	1006	0.461	976	924	0.447
0.315	1508	0.531	1585	1494	0.533
0.285	2024	0.592	2127	1892	0.570
0.249	3003	0.726	3403	2586	0.645

The post-fire measured burn rates were within good agreement of the predictions for both the the baseline aluminized HTPB and SAA-157 motors.

The results of test data in Tables 5 and 6 are shown graphically in Figure 5. As expected, the metal/metal oxide containing SAA-157 propellant exhibited a higher burn rate than the baseline aluminized HTPB. In addition, the slope break at approximately 1900 psi seen in the baseline aluminized HTPB propellant was pushed out by the burn rate increase in SAA-157 to past the highest pressure measured for the ballistic motors. This shift in the pressure exponent slope break is expected to be helpful in rocket motor design at operating pressures slightly higher than with the baseline aluminized HTPB propellant.

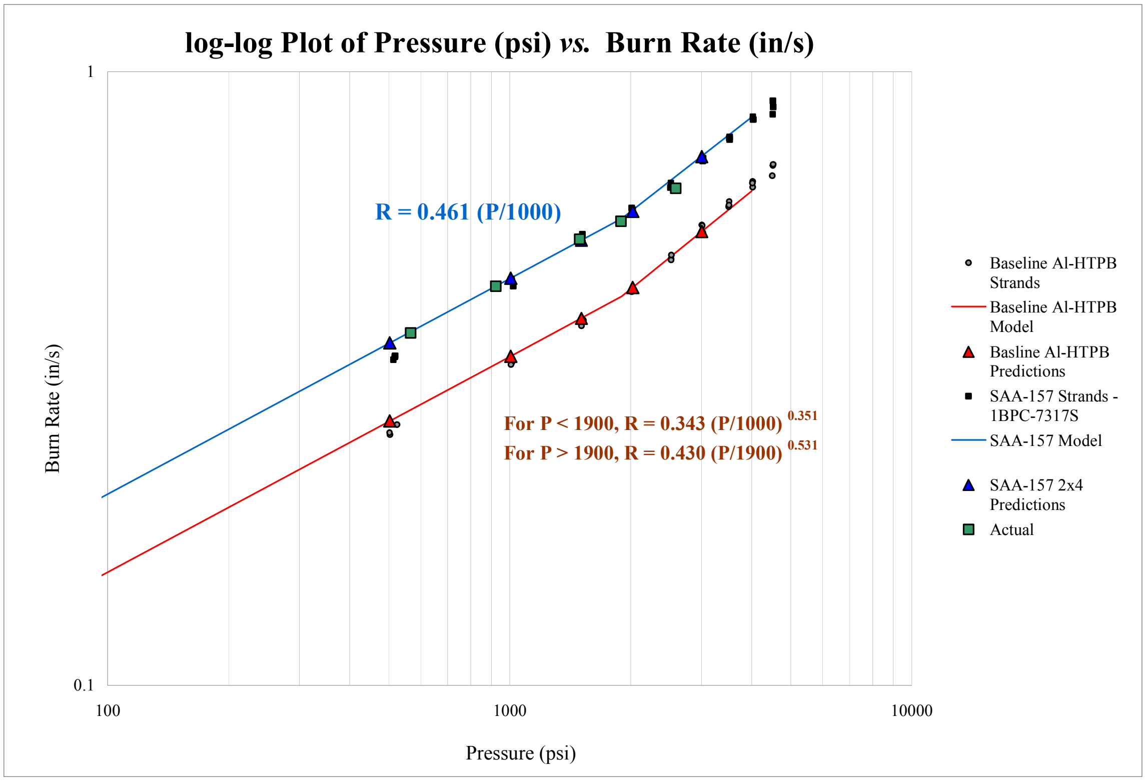

Fig. 5. Ballistic Models from Sub-scale Ballistic Evaluation Motor Static Firing.

Concluding remarks

Nanocomposite $4Al{\cdot}Fe_2O_3$ powder prepared by ARM is evaluated as a burn rate modifier for HTPB propellants. Current tests established compatibility of the new material with the current propellant ingredients and showed that the propellant formulations prepared with this additive pass all necessary mechanical and safety evaluations. Strand burning tests as well as static subscale ballistic tests showed substantial increase in the propellant burn rate when the burn rate modifier is added to the formulation. Furthermore, the slope break in the log of burn rate vs. log of pressure curve is shifted to a higher pressure for the formulation with the nanocomposite modifier. This shift is expected to be advantageous for the design of rocket motors operating at pressures higher than those used for the current high performance aluminized HTPB propellants.

Acknowledgement

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